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Pesticide Assessment Guidelines Subdivision O Addendum 2, Residue Chemistry. Series 171-4 Analytical Methods(s), Magnitude of the Residue Crop Field Trials and Storage Stability Study

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PESTICIDE ASSESSMENT GUIDELINES,

SUBDIVISION O,
Addendum 2,
RESIDUE CHEMISTRY

Series 171-4,

Analytical Method(s),

Magnitude of the Residue: Crop Field Trials

and

Storage Stability Study

Prepared by:

M.J. Nelson, Ph.D. and F.D. Griffith, Jr.
Residue Chemistry Branch
Hazard Evaluation Division

Project Manager:

Elizabeth M.K. Leovey, Ph.D
Hazard Evaluation Division

Office of Pesticide Programs
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16. Abstract (Limit: 200 words)

The Data Reporting Guideline (DRG) for residue chemistry for Analytical Methods, Magnitude of the Residue: Crop Field Trials, and Storage Stability Study, Series 171-4, gives guidance to pesticide registrants on the format for their study report so that the Agency can review it efficiently. This guidance clarifies sections in the existing Pesticide Assessment Guidelines, Subdivision O, on data reporting. It provides an outline for the study report listing items which should be discussed. Data submitters can use the DRG in preparing their reports for submission to EPA to meet 40 CFR 158 data requirements for the registration of pesticides.

The DRG for Series 171-4, is only one of a number of DRG's published by the National Technical Service as an addendum to Subdivision O of the Pesticide Assessment Guidelines.

17. Document Analysis a. Descriptors

b. Identifiers/Open-Ended Terms

c. COSATI Field/Group

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Subdivision O - Analytical Method(s)

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PESTICIDE ASSESSMENT GUIDELINES

RESIDUE CHEMISTRY

Analytical Method(s)

Subdivision O, Series 171-4

DATA REPORTING

INTRODUCTION

A. Purpose

To provide suitable analytical methodology for residue data gathering and for tolerance enforcement purposes.

A complete copy of each method should be submitted. The copy submitted should be non-confidential. Each method should be a separate report. The description of the analytical method(s) should be sufficiently detailed so that it can be repeated in EPA's laboratories. Analytical methodology may also be required for metabolites. The company should indicate the method preferred for regulatory purposes on specific commodities. Registrants are reminded of requirements delineated in 40 CFR 158.125(b)(15) for multiresidue method testing.

B. Objective

This Data Reporting Guideline is designed to aid the petitioner/registrant in generating reports which are compatible with the Agency's review process. While following this Guideline is not mandatory, data submitters are encouraged to submit complete reports which can be efficiently reviewed by the Agency.

RESPONSE TO PUBLIC COMMENTS

The purpose of this section is to acknowledge and address the concerns expressed in the letters of comment received by the Agency in response to the public notice in Federal Register Volume 50, No. 147, p. 31010, July 31, 1985.

This addendum to the Pesticide Assessment Guidelines (Subdivision O) is to be considered an all-encompassing document. EPA recognizes there are sections in the addendum which do not apply in all cases. Therefore, registrants should exercise scientific judgment in deciding which portions are germane to a specific data submission.

This Data Reporting Guideline is not intended to introduce new data requirements or revisions into the Pesticide Assessment Guidelines (Subdivision O); nor is it intended for use by Agency data reviewers as a mere checklist. It is intended to clarify ambiguities in interpretation of those existing Guidelines, and to organize the submission of data to facilitate the review process.

For comments relating to Good Laboratory Practices (GLPs), the Agency has revised its requirement in this document in recognition that there is currently no regulatory GLP Guideline for chemistry data.

GUIDELINE

TITLE/COVER PAGE

Title page and additional documentation requirements (i.e., requirements for data submission and procedures for claims of confidentiality of data) if relevant to the study report should precede the content of the study formatted below. These currently proposed requirements are described in 49 FR (188) 37596 (9/26/84).

TABLE OF CONTENTS

I. SUMMARY/INTRODUCTION

A. Scope (suitable matrices) and source of method (e.g. Pesticide Analytical Manual, company reports, etc.); and

B. Principles of the analytical procedure (describe) including identification of the chemical species determined and the limits of detection and sensitivity.

II. MATERIALS/METHODS

A. Equipment (list and describe);

B. Reagents and standards (list and describe source and preparation);

C. Analytical procedure (detail in a stepwise fashion, with special emphasis on reagents or procedural steps requiring special precautions to avoid safety or health hazards):

1. Preparation of sample;

2. Extraction [demonstrate efficiency, if relevant (e.g., dry crop substrates, "bound" residues, etc.)];

3. Fortification, if applicable (i.e., during method validation runs];

4. Clean-up; and

5. Derivatization (if any).

D. Instrumentation (to include information on]:

1. Description [e.g., make/model, type/specificity of detector(s), column(s) (packing materials, size), carrier gas(es), etc.];

2. Operating conditions [e.g., flow rate(s), temperature(s), voltage, etc.]; and

3. Calibration procedures.

E. Interference(s) (describe tests):

1. Sample matrices;

2. Other pesticides;

3. Solvents; and

4. Labware.

F. Confirmatory techniques (describe);

G. Time required for analysis (to carry a sample/set completely through the analytical procedure, including the determinative step);

H. Modifications or potential problems, if any in the analytical method(s) (detail circumstances and corrective action to be taken);

I. Methods of Calculation (describe in a stepwise fashion):

1. Calibration factors; and

2. Analyte in sample.

J. Other (any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of residue analytical methodology and the means of calculating the residue results).

III. RESULTS/DISCUSSION (describe expected performance of method)

A. Accuracy (expected mean and range of recoveries);

B. Precision;

C. Limits of detection and quantification (provide definition);

D. Ruggedness testing, if performed; and

E. Limitations.

IV. CONCLUSION

Discuss applicability of analytical procedure for measuring specific test compound(s) in various test substrate(s), ready availability of equipment, interference(s), etc.

V. CERTIFICATION

Certification of authenticity by the study director (including signature, typed name, title, affiliation, address, telephone number, date).

VI. TABLES/FIGURES

VII. REFERENCES

VIII. APPENDIX(ES)

A. Representative chromatograms, spectra, etc. (as applicable); and

B. Other (any relevant material not fitting in any of the other sections to this report).

Subdivision O - Magnitude of the Residue: Crop Field Trials

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PESTICIDE ASSESSMENT GUIDELINES

RESIDUE CHEMISTRY

Magnitude of the Residue: Crop Field Trials[1/]

Subdivision O, Series 171-4

DATA REPORTING

INTRODUCTION

A. Purpose

Crop field trials to provide residue chemistry data on the magnitude of the residue are required by 40 CFR 158.125 to support the registration of any pesticide intended for use on a food or feed crop under the amended Federal Insecticide, Fungicide, and Rodenticide Act.

Residue chemistry data on raw agricultural commodities (r.a.c.) are used by the Agency to estimate the exposure of the general population to pesticide residues in food, and for setting and enforcing tolerances for pesticide residues in or on raw agricultural foods or feeds under provisions of Section 408 of the Federal Food, Drug, and Cosmetic Act. (Note: Processed foods and feeds are regulated under Section 409 of the Act.)

Residue chemistry data are also needed to support the adequacy of one or more methods for the enforcement of the tolerance, and to support practical methods for removing residues that exceed any proposed tolerances.

Subdivision O (Residue Chemistry) of the EPA Pesticide Assessment Guidelines (§§ 171-3 and §§ 171-4) and the Guidelines on Pesticide Residue Trials developed under the auspices of the Codex Committee on Pesticide Residues (FAO Plant Protection Bulletin, 29:1/2, pp. 12-27, 1981) provide information to aid the petitioner/registrant in conducting crop field trials.

1/ Other aspects of the Magnitude of the Residue (see 40 CFR 158.125: processed food/feed; meat/milk/poultry/eggs; dermal uses; fumigation uses; etc.) will be addressed in separate Data Reporting Guideline documents.

B. Objective

Crop field trial studies should answer the following question: What is the maximum level of "total toxic residue" that will likely result in or on the raw food or feed commodity as a result of application of the pesticide formulated product according to the proposed label directions for use?

The term "total toxic residue" is used to describe the sum of the parent pesticide and its degradation products, metabolites (free or bound), and impurities that are considered to be of toxicological significance, and therefore warrant regulation.

Actual residue data on commodities as consumed should be provided in cases where tolerance level residues lead to unreasonable risks.

Crop grouping considerations should also be addressed, and an effort made to achieve harmonization with applicable International Residue Limits (Codex).

This Data Reporting Guideline is designed to aid the petitioner/registrant in generating reports which are compatible with the Agency's review process. While following this Guideline is not mandatory, data submitters are encouraged to submit complete reports which can be efficiently reviewed by the Agency.

RESPONSE TO PUBLIC COMMENTS

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GUIDELINE

The petitioner/registrant's report on crop field trials on a raw agricultural commodity should include all information necessary to provide a complete and accurate description of field trial treatments and procedures; sampling (harvesting), handling, shipping, and storage of the r.a.c.; storage stability validation of the test chemical [and metabolite(s) of toxicological concern] in a plant matrix; residue analyses of field samples for the "total toxic residue" (and for individual components of toxicological concern); validation (recovery studies) of the residue analytical methodology; reporting of the data and statistical analyses; and, quality control measures/precautions taken to ensure the fidelity of these operations. The following is the suggested format for the report.

[Note: The Analytical Method(s) and Storage Stability formats are addressed in separate Data Reporting Guideline documents being developed concurrently.]

MASTER COVER PAGE

Title page and additional documentation requirements (i.e., requirements for data submission and procedures for claims of confidentiality of data) if relevant to the study report should precede the content of the study formatted below. These currently proposed requirements are described in 49 FR (188) 37596 (9/26/84).

MASTER TABLE OF CONTENTS

I. Master Introduction and Summary
II. Master Data Table(s) and Other Graphic Presentation(s)
III. Individual Field Trial Study Reports, including Analytical Method(s), Method(s) Validation, Storage Stability, and Statements of Authenticity
IV. Certification
V. References
VI. Appendixes (list contents)

I. MASTER SUMMARY/INTRODUCTION

A. Purpose of studies [include target pest(s)/disease(s)];

B. Results [including explanations for apparently aberrant, atypical values, or outliers; discussion of geographical representation (major growing areas), seasonal variation (summer/winter, wet/dry, etc.), and representativeness of types and varieties of the r.a.c.; and whether the intended use is claimed to be a "minor use" (ref. 40 CFR 158.60; EPA's policy concerning data requirements for minor uses of pesticides includes the following elements: a crop which is planted on a small total amount of acreage or a use which is otherwise limited such that the potential market volume of the product for that use is inherently small, thus resulting in the risk and exposure associated with the proposed use being correspondingly low. For further discussion, see the EPA Policy Statement on the Minor Uses of Pesticides, OPP-36114.)];

C. Field procedures;

D. Analytical procedures/instrumentation;

E. Method recovery validation data;

F. Storage stability;

G. Discussion [including Quality Control measures taken; statistical treatment(s) of data; and information on the level(s) of the "total toxic residue" (including any individual component(s) of the residue of special concern) in or on the r.a.c. (specific plant part(s)) arising from the use of the pesticide formulated product on the test crop under specific use conditions. Results should also be correlated to the storage stability study.]; and

H. Conclusions.

II. MASTER DATA TABLE(S) AND OTHER GRAPHIC PRESENTATION(S)

A. Summary map (USA, include outside USA, if applicable) of crop field study sites (by crop);

B. Summary table(s) of residue results of individual field trials;

C. Graphic representation(s) (e.g., residue decline), figures, flowcharts, etc.;

D. Summary table(s) of recovery data via the analytical methodology;

E. Summary table(s) of storage stability validation data;

III. INFORMATION/RAW DATA ON INDIVIDUAL FIELD TRIALS (specifically, each individual field trial report should include the following information)

A. Test Substance (Pesticide)

1. Identification of the test pesticide active ingredient (a.i.), including chemical name, common name (ANSI, BSI, ISO), and company developmental/experimental name;

2. Identification of the pesticide formulated product(s) used in the field trial, including trade name, type (EC, WP, G, etc.), and amount of active ingredient per gallon, pound, etc., EPA registration number (if available), manufacturer, and check of composition (% a.i.) prior to its field use;

3. Information on other relevant parameters, as pertinent, e.g., tank mate(s), spray additive(s), carrier (encapsulating polymer, etc.), etc.; and

4. Other (any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of the test chemical).

B. Test Commodity (r.a.c.)

1. Identification of the r.a.c., including type/variety and crop group classification [40 CFR 180.34(f), as revised 6/29/83, 48 FR 29855];

2. Identification of specific crop part(s) harvested; used in residue analytical methodology validations; and subjected to residue analysis for a determination of the "total toxic residue";

3. The developmental stage(s), general condition (immature/mature, green/ripe, fresh/dry, etc.), and size(s) of the r.a.c. at time of pesticide application(s) and at harvesting(s); and

4. Other (any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of the r.a.c.).

C. Test Procedures

1. A detailed description of the experimental design and procedures followed in the growing of the r.a.c., application(s) of the pesticide formulated product(s), and harvesting(s) of samples. The information provided, which may be presented on standardized field sheets, should include (in addition to a description of the test substance and test commodity):

   a. Trial identification number;

   b. Cooperator (name and address), test location (county and state - country, if outside U.S.A.), and year;

   c. Field trial lay-out (e.g., size and number of control and experimental plots, number of plants per plot/unit area, number of rows per plot, length of rows and row spacing);

   d. Cultural treatment(s) [farming practice (cultivation, irrigation, etc.) and cropping system];

   e. Soil characteristics (name/designation of the soil type and its physical and chemical properties, including pH and percent organic matter);

   f. Method(s) of application (air or ground) of the pesticide formulated product(s), description of the application equipment, type of application (band/broadcast, soil/foliar/directed, ULV/concentrate/dilute, other), and, calibration of pesticide application equipment, including methods and dates;

   g. Dose rate(s) (amount of active ingredient and formulated product per acre, row, volume, etc.) and spray volume(s) (per acre, etc.);

h. Number and timing of application(s) [total number, during dormancy, preplant, preemergence, prebloom, etc., between-application-interval(s), and treatment-to-sampling interval(s) (aka TSI or PHI)];

i. Other pesticide(s) applied [identity (name and type of formulated product(s), active ingredient(s)), rate(s), date(s), tank-mate or separate, purpose of use];

j. Climatological data (record of temperatures and rainfall during the growing season from the nearest weather station, and wind speed during applications);

k. Dates [planting/sowing/transplanting, as applicable, other significant dates in the growing of the crop (e.g., husk split for tree crops), pesticide application(s), harvest(s)];

l. Harvest procedures [method of harvesting (mechanical/hand, from the plant/ground/flotation, etc.), type equipment used, number/weight of samples collected per replication and number of replications per treatment level, sample coding (cross-referenced to sample history), etc.];

m. Quality control (control measures/precautions followed to ensure the fidelity of the crop field test); and

n. Other [any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of the growing of the r.a.c., application(s) of the pesticide formulated product(s), and harvesting(s) of samples].

2. A detailed description of the handling, pre-shipping storage, and shipping procedures for harvested r.a.c. samples. The information provided, which may be presented on a standardized form, should include (in addition to a description of the test substance and the test commodity):

a. Sample identification (means of labeling/coding);

b. Conditions [temperatures, container type(s)/size(s), sample size(s), etc.] and duration of storage before shipping;

c. Method(s) of packaging for shipment [container type(s)/size(s), sample size(s), ambient/iced, labeling/coding, etc.];

d. Means of transport from the field to the laboratory;

e. Dates (harvest, pre-shipping storage, shipping, and receipt in the laboratory);

f. Quality control (control measures/precautions followed to ensure the fidelity of harvested samples during handling, pre-shipping storage, and shipping operations); and

g. Other (any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of the handling, preshipping storage, and shipping procedures for harvested samples).

3. A detailed description of the conditions and length of storage of harvested r.a.c. samples following their receipt in the laboratory.

Refer to the Data Reporting Guideline for a Storage Stability Study[2/] for guidance on this subject area.

4. A detailed description of the residue analyses used in determining the "total toxic residue" in r.a.c. field trial and storage stability samples. (Note: If the specified information is provided elsewhere within the overall data submission package, it need not be reiterated here. In that case, a reference to the relevant analytical methodology would be sufficient.)

Refer to the Data Reporting Guideline on Analytical Method(s)[2/] for guidance on this subject area.

5. Method recovery validation studies should be run concurrently with the residue analyses of crop field trial samples from each individual field trial in order to provide information on the recovery level(s) of the test compound(s) from the test substrate(s) at various fortification level(s) using the residue analytical methods, and to establish a validated method sensitivity. The following information specific to the method validations, which may be presented on a standardized form, should include:

a. Experimental design [e.g., identity of test substrate(s) (specific plant part(s)) and test compound(s) (parent/specific metabolite(s)), number and magnitude of fortification levels, number of replicate samples per test compound per fortification level, sample coding, control samples, etc.];

b. Fortification procedure [detail the preparation of the test compound(s) and test substrate(s) and the manner in which the test compound(s) was/were introduced to the test substrate(s)];

c. Dates [test sample preparation (maceration/extraction/etc.), test compound(s) preparation (standard solution(s) of known concentration), residue analyses];

2/ Concurrently in preparation.

d. Residue results [raw data, ppm theoretical/found, procedure(s) for calculating percent recoveries, recovery levels (range), sensitivity and limit of detection]; and

e. Other (any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of analytical methodology validation procedures).

D. Organization of Data Tables and Forms

1. Table(s) of residue assay data for specific plant parts analyzed;

2. Table(s) on residue recovery values;

3. Graph(s), as pertinent (e.g., residue decline);

4. Form(s) containing field trial history information;

5. Form(s) containing harvesting, shipping, storage information;

6. Table(s) of weather data.

IV. CERTIFICATION

A signed and dated certification of authenticity by, and identifying information (typed name, title, affiliation, address, telephone number) on, the personnel responsible for the various phases of this report (e.g., Study Director, Field Supervisor, and Laboratory Supervisor).

V. REFERENCES

VI. APPENDIX(ES)

A. Representative chromatograms, spectra, etc. of reagent blanks, solvent blanks, reference standards, controls, field samples, fortified samples, etc. (cross-referenced to individual field trial study reports);

B. Reprints of published and unpublished literature, company reports, letters, analytical methodology, etc. cited (or used) by the petitioner/registrant (unless physically located elsewhere in the overall data report, in which case cross-referencing will suffice);

C. Other (any relevant material not fitting in any of the other sections of this report).

Subdivision O - Storage Stability Study

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PESTICIDE ASSESSMENT GUIDELINES

RESIDUE CHEMISTRY

Storage Stability Study

Subdivision O, Series 171-4

DATA REPORTING

INTRODUCTION

A. Purpose

To provide information on the effects of storage on the magnitude of the residue in a raw agricultural commodity (r.a.c.) matrix.

All of the data/information necessary to independently verify the results and conclusions drawn from the data results, etc., should be included.

Separate reports using the following guidance should be prepared for each study performed.

A storage stability study on a different r.a.c. will be considered relevant only if that r.a.c. is a related crop and if the experimental design is appropriate to current considerations.

B. Objective

This Data Reporting Guideline is designed to aid the petitioner/registrant in generating reports which are compatible with the Agency's review process. While following this Guideline is not mandatory, data submitters are encouraged to submit complete reports which can be efficiently reviewed by the Agency.

RESPONSE TO PUBLIC COMMENTS

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TABLE OF CONTENTS

I. SUMMARY/INTRODUCTION

- Purpose
- Results (include summary table of storage validation data)
- Sample preparation and fortification
- Storage and sampling procedures
- Analytical procedures
- Methods of calculation

II. MATERIALS

A. Test Substance

1. If fortification used, describe the test substance(s) [chemical/common/experimental/CAS name(s), including the determination/check of the purity of the test compound(s) (parent plus any metabolites(s) of special concern, all in reference standard form) and preparation of standard solutions];

2. If weathered residue sample used, identify the nature and amount of test substance(s) therein at "zero time" (defined as the beginning of the storage stability testing); and

3. Other [any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description and identification of the test substance(s) used in storage stability validation testing].

B. Test Commodity

1. Identification of the r.a.c.(s) (crop/type/variety) and the specific crop part(s) to be used in storage stability testing;

2. The developmental stage(s), general condition (immature/mature, green/ripe, fresh/dry, etc.) and size(s) of the r.a.c. samples used in storage stability testing;

3. Treatment/preparation of r.a.c. sample(s) prior to storage stability testing [e.g., trimming, cleaning, or other means of residue removal, compositing, subsampling, chopping, extraction, etc., refer to the FDA Pesticide Analytical Manual (P.A.M.), Volume I, §§ 141-142 for recommended procedures];

4. Sample identification [source of sample(s), field trial identification number, control or weathered residue sample, coding and labeling information (should be the same as, or cross-referenced to, the sample coding/labeling assigned at harvest];

5. Other [any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of the r.a.c.(s)].

III. METHODS

A. Experimental design (e.g., number of test commodities, number of test substances, number and magnitude of test levels, number of replicate samples per test compound per test level, number of sampling intervals, representativeness of test commodities to the matrices of concern, etc.)

B. Test Procedures

1. Fortification ("spiking") procedure, if used [detail the manner in which the test compound(s) was/were introduced to the test substrate(s)];

2. Storage conditions [temperature, humidity, lighting, container type(s)/size(s), crop form (extract/macerate/etc.), sample size(s)/weight(s), duration; etc.];

3. Sampling (at zero time and at regular intervals thereafter, describe sampling procedure, duration of study should correspond to the length of storage of the field trial samples collected for residue analysis);

4. Dates [sample preparation (maceration/extraction/etc.), "spiking" or determining the type/amount of weathered residue (zero time), periodic sampling intervals, end of storage, residue analyses];

5. Method(s) of residue analysis:

a. Title/designation/date and source [Pesticide Analytical Manual (P.A.M.), Volume II, scientific literature, company reports, etc.), or cross-reference Analytical Method section of submission if same method(s) used];

b. Discuss any deviations (in reagents, procedures, instrumentation, operating parameters, etc.) from the Analytical Method(s) used for residue analysis of field trial samples, if same method(s) used; otherwise,

c. Detail the principles and stepwise procedures (extraction/clean-up, derivatization, determination), including any modification(s) made, chemical species determined, confirmatory techniques used, if any, etc., extraction efficiency (if pertinent);

d. Instrumentation and operating parameters [make/model, type/specificity of detector(s), column(s) (packing materials, size), carrier gas(es), flow rate(s), temperature(s), voltage, limit of detection and sensitivity, calibration procedures, etc.];

e. Reagents or procedural steps requiring special precautions (to avoid safety or health hazards, explain);

f. Time required for analysis (to carry a sample/set completely through the analytical procedure, including the determinative step);

g. Procedure(s) for calculating residue level(s) and percent recoveries (detail); and

h. Other (any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of the analytical methodology and the means of calculating the residue results).

IV. RESULTS/DISCUSSION

A. Residue results [raw data, dilution factor(s), peak heights/areas, method correction factor(s) applied, formula(e)/standard curve(s) used, ppm theoretical/found, recovery levels (range), percent recovery vs. length of storage (dissipation data), appropriateness of length of storage study, etc.];

B. Statistical treatment(s) [describe test(s) applied to the raw data];

C. Quality control [if not covered elsewhere: control measures/precautions followed to ensure the fidelity of storage stability validation(s)]; and

D. Other (any and all additional information the petitioner considers appropriate and relevant to provide a complete and thorough description of storage stability validation results).

V. CONCLUSION

Discuss conclusions that may be drawn regarding the stability of the test compound(s) in the test matrices as a function of storage time.

VI. CERTIFICATION

Certification of authenticity by the Study Director (including signature, typed name, title, affiliation, address, telephone number, date).

VII. TABLES/FIGURES

A. Table(s) of raw data from storage stability validation testing;

B. Graphs, figures, flowcharts, etc. (as relevant).

VIII. REFERENCES

IX. APPENDIX(ES)

A. Representative chromatograms, spectra, etc. (as applicable);

B. Reprints of methods and other studies cited (unless physically located elsewhere in the overall data submission, in which case cross-referencing will suffice); and

C. Other (any relevant material not fitting in any of the other sections to this report).